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WADD-TR-61-94
PART II

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TOTAL NORMAL AND TOTAL HEMISPHERICAL EMITTANCE OF POLISHED METALS – PART II

TECHNICAL REPORT NO. WADD-TR-61-94, Part II

January 1963

Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 7360, Task No. 736001

(Prepared under Contract No. MIPR 33(616)-61-7
by the U. S. Naval Radiological Defense Laboratory,
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20-457C, 700, 2-27-63

FOREWORD

This report was prepared by the U. S. Naval Radiological Defense Laboratory under MIPR 33(616)-61-7. This contract was initiated under Project No. 7360, "The Chemistry and Physics of Materials," Task No. 736001, "Thermodynamics and Heat Transfer." It was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Mr. Raymond Prezecki acting as project engineer.

ABSTRACT

The total hemispherical emittance, the total normal emittance, the spectral emittance at 0.65 microns and the electrical resistivity were measured on freshly rolled and polished surfaces of molybdenum in a vacuum over a temperature range from 1100°K to 1800°K. The total hemispherical emittance was obtained from the measured power dissipation at the center of an electrically heated strip suspended in a vacuum while its temperature was measured by a thermocouple. The total normal emittance was measured using a radiation thermopile. The ratio of total hemispherical to total normal emittance was also calculated directly from the angular distribution of radiation obtained by revolving the detector about an axis through the ribbon.

Observations were also made on the time variation of the total hemispherical emittance of platinum. Small increases were noted in the emittance while thermal etching of the boundaries between the crystals was followed by time lapse photomicroscopy of the heated surface.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

J. I. WITTEBORT
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INTRODUCTION

The research covered in this report is a continuation of that presented in WADD-TR-61-94 (Total Normal and Total Hemispherical Emittance of Polished Metals).[1] The background material discussed in the previous report along with the details of the experimental apparatus and techniques are pertinent to the material covered in this volume.

This year's work included three phases: (1) Measurements of the total hemispherical emittance, total normal emittance, angular distribution of the total emittance, and the spectral emittance at 0.65 microns of freshly rolled and polished strips of molybdenum between 1100°K and 1800°K. The experimental apparatus was essentially the same as that of the previous report. (2) The design and construction of a new and improved emittance measurement system incorporating a higher operating temperature capability, greater reliability, lower chamber pressure, and a rotating specimen mount. This rotation will facilitate some spectral emittance measurements which will assist in the interpretation of the total emittance data. (3) Continuation of the total hemispherical emittance and spectral emittance at 0.65 microns versus time measurements on platinum. These measurements are correlated with a series of photomicrographs of the surface as the thermal etching and recrystallization proceed.

The most important parameter of radiant heat transfer is the total hemispherical emittance which is defined as the ratio of the total radiation emitted from a unit area to that emitted by an equal area of a perfectly black surface at the same temperature regardless of angle. This emittance is a function of the bulk material, the condition of the surface and the temperature. Since the total normal emittance which considers only the total radiation in a small solid angle at the surface normal is the parameter most often measured; it is of interest to investigate the relationship between the total hemispherical emittance and the total normal emittance for various classes of materials. This is accomplished on this project by measuring both parameters for the same surface as a function of temperature and checking this result with angular distribution measurements of the total radiation.

Since the accuracy of a temperature measured with an optical pyrometer depends upon a reliable value of the spectral emittance at 0.65 microns, the spectral emittance as a function of temperature and its time variation is included for most of the surfaces studied on this project.

While a polished surface does not remain polished at high temperature due to recrystallization and thermal etching, it is reasonable to expect that an equilibrium condition would be reached after the surface is held at a fixed temperature for a sufficiently long period of time. When this condition is established the size distribution of the exposed crystallite faces and the width and depth of the boundaries should be constant,

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[1] Manuscript released by the authors December 1961 for publication as a WADD Technical Report.

although individual grains might continue to expand or contract. From a practical point of view, the final emittance of this equilibrated surface is of the most importance at high temperatures. The rate at which the surface proceeds from the polished to the equilibrium state is an increasing function of the temperature. At low temperatures below approximately 0.4 times the absolute temperature of melting, the surface changes would be extremely slow and the emittance of the polished surface would be the most significant. One goal of phase 3 is to investigate the rate at which the emittance changes as a function of temperature and the relationship between the final emittance and the emittance of the polished surface. In pursuing this goal, it is necessary to investigate the effect of the prior temperature and mechanical history on the value of the final emittance.

EMITTANCE VERSUS TEMPERATURE

The apparatus used for the molybdenum studies was essentially the same as that used for platinum[1] except for a higher capacity power supply and the addition of water cooling to the thermopile housing and the bell-jar. The thermopile cooling was accomplished by soldering 1/8 in. copper tubing to the outside of the copper heat sinks, item (A) of Fig. (1) in the last report.[1] This tubing was connected to flexible Tygon tubing to allow rotation of the thermopile mount. This cooling reduced the drift of the thermopile considerably. Cooling the bell-jar with 1/4 in. copper tubing fastened to the outside surface with heat conducting cement reduced outgassing of the internal walls resulting from the absorption of radiation from the ribbon. The power supply was capable of delivering 0 to 70 VAC RMS at 10 KW with voltage regulation to ± 1 per cent. Even though the power supply had this capability, the remaining part of the power delivery and heat dissipation system precluded any extended measurements above 1600°K.

A new system shown in Fig. (1) is virtually complete and it has been designed so that ribbon temperatures up to at least 3000°K can be realized. The 10 KW power supply system will be used as well as the water cooled bell-jar. In this case, the thermopile mount is stationary and the ribbon rotates 230° about its axis. The ribbon clamps and exposed supporting structure are water cooled. In addition, an improved method for cooling the thermopile cold junctions is employed. A water cooled background, consisting of a blackened brass plate behind the ribbon forms the thermopile's total field of view (except for the ribbon). This plate with horizontal 30° grooves 1/16 in. deep machined in the surface is the black rectangle in Fig. (1). Improvements in the vacuum system include increased capacity and a liquid nitrogen cold trap within the bell-jar. Tungsten/tungsten - 26 per cent rhenium thermocouples are on hand for measurement of temperatures from 1000°C to 2800°C.

The techniques previously used to measure the temperature of the platinum ribbons were found to be unsatisfactory when applied to molybdenum. Formerly, the thermocouple wires were spot welded to the ribbon. After considerable effort, it was found impractical to obtain a firm weld of 0.005 inch platinum/platinum - 10 per cent rhodium thermocouple wires to a molybdenum ribbon. This was probably due to the wide separation

in the melting points of the wire and the molybdenum sample making the right combination of welding parameters difficult to achieve. Consequently, a mechanical method similar to riveting was developed for attaching the wires. This involved drilling a 0.0059 inch hole through the molybdenum into which was inserted the thermocouple wire so that the end of the wire protruded through the ribbon by about one wire diameter. This was done by gripping the wire in a miniature collet, placing the ribbon on the collet top with the wire through the hole and then clipping the wire with surgical scissors. The protruding end is then tapped with a small drift punch, spreading the wire on both sides of the ribbon. The resulting connection was found to be electrically and mechanically secure up to the melting point of the thermocouples. A much smaller temperature depression near the thermocouple wire contact was observed with this type of attachment.

Six different samples of molybdenum were examined in the current study. All six were obtained from the same supplier but in two different batches. Sample number 1 was part of a six foot ribbon with a fairly specular surface as received. Samples number 2, 3 and 4 were three of six 12-in. pieces, and did not appear as specular to the eye as did number 1. Samples number 1 and 2 were studied in the condition as received except for degreasing with acetone, cleaning with a rubber eraser and wiping with acetone. Photomicrographs of these specimens before and after heating are shown in Fig. (2). Sample number 3 was polished, using rouge in wax, on a buffing wheel attached to a horizontal milling machine. Polishing was continued until the surface showed no apparent change. This particular surface appeared more diffuse than in the as received condition, and upon microscopic inspection, the grain boundaries were well defined, as can be seen in Fig. (3). These grains are probably the particles from the powder metallurgy forming process.

Sample numbers 4, 5, and 6 were polished on a circular rotatable drum that could be lowered to the surface of a rotating lap. The axis of the drum and lap intersected at right angles. In this way the lap, which rotated at high speed, contacted the ribbon over a small area; the whole ribbon being polished by rotating the drum slowly. The resulting polish, using fine aluminum oxide powder, was very specular. A photomicrograph of one of these surfaces appears in Fig. (3).

In order to determine the electrical resistivity of the sample under test, it is necessary to know the cross sectional area to a reasonable accuracy. There are three ways of determining this area. First, knowing the length and width of the ribbon plus the density, the area may be computed from its measured weight. Second, the area may be computed from direct measurement of its width and thickness, and third, if the electrical resistivity at room temperature is known, the average cross sectional area between two points on the ribbon may be determined from a measurement of the resistance between these points. In the first case, the density of molybdenum particles formed by powder metallurgy is apt to be different for different samples, putting a limitation on this technique. The second method involves a rather precise measurement of the thickness which will vary slightly

from point to point along the ribbon. A micrometer has been found to give thicknesses about 5 to 10 per cent higher than the average electrical cross sectional area. This is most likely due to surface irregularities. The third method has been adopted as the most reproducible even though it requires a knowledge of the electrical resistivity at room temperature, a number that varies from source to source. However, it appeared that a value of 5.17×10^{-6} ohm-cm at $0^{\circ}C$ is the most frequently quoted number. Using this value and a temperature coefficient of resistivity of 0.0033 per degree, the resulting cross sectional area was computed from the measured resistance between the two voltage probes about 7 cm apart on the ribbons.

In case the thermocouples became unreliable when maintained near their melting points for extended times, a resistivity versus temperature curve was established, using data obtained rapidly, from which the temperature could be determined at later times. This curve is displayed in Fig. (4). However, the resistance of the molybdenum also underwent an increase with time in all four samples. Sample number 1 changed most drastically. In fact, it became impossible to interpret the results at $1500^{\circ}K$ because of a rather rapid increase. The measurement was terminated and a spectroscopic analysis performed on the ribbon as a check on any impurities that could react with residual gases within the vacuum system. The analysis was inconclusive. The thickness, as measured with a micrometer, increased approximately 5 per cent contrary to what an increase in resistivity would predict. This phenomenon has not been explained. Sample numbers 2 and 3 exhibited some increase in the measured resistivity with time at the higher temperatures which was probably due to surface evaporation. The change was small enough to have little effect on the smoothed resistivity data as displayed in Fig. (5). However, when the temperature is determined for use in calculating emittance, any error is magnified due to the fourth power relationship of temperature. Figure (5) is the composite of all resistivity data from samples number 1, 2 and 3 where the separate curves for each sample have been normalized to the curve in Fig. (4) by adjusting the value of the cross-sectional area. This curve in Fig. (5) is nearly identical to that obtained by Armour.[2]

The data reduction techniques used in evaluating the emittance data have been discussed at considerable length in the last report.[1] Figure (6) is the plot of total hemispherical and total normal emittance for the first three samples. It should be noted that during the various changes in electrical resistivity the emittance remained quite stable except for sample number 2 after extended aging. The total normal emittance was determined from the total hemisperical emittance and the angular distribution measurements. On each of these three samples, post heating inspection revealed a thin bluish-white film in various amounts, depending on the temperature-time history. In order to qualitatively study the behavior of this film, sample number 4 was polished as explained before and emittance measurements made.

When the ribbon was initially raised to $1100^{\circ}K$, the power supplied to the ribbon necessary to maintain a constant temperature increased rapidly at first and then asymptotically to some equilibrium value. Calculation of the total hemispherical emittance based on this equilibrium

power yields a value identical to the previously obtained emittance of approximately 0.198 at 1100°K. Due to the rapid change (overall about 10 minutes) which was not expected in this vacuum of between 10^{-4} and 10^{-5} Torr, the initial value of emittance was not obtained; however, a rough estimate put it near the theoretical curve in Fig. (6). After about five minutes, a value of approximately 0.142 was observed for the total hemispherical emittance. Upon cooling, it was noted that the resistivity had undergone an insignificant change; subsequent heating to higher temperatures yielded emittance values identical to those previously obtained up to 1700°K. Above this temperature, the emittance deviated from this curve so that at 1900°K a value about 2 or 3 per cent less was noted. Returning to 1100°K, the emittance was found to be about 6 per cent less than the initial equilibrium value; however, after about 5 minutes, the emittance climbed back up to about 0.198. Visual observation of the ends of the cold ribbon show various amounts of the whitish film in the region where the ribbon temperature varied in some fashion from the support temperature to the highest in the center (1900°K). It appears that above some intermediate temperature, the film becomes thinner. At this time, it was felt that the formation of the film was a direct cause of the increasing emittance and that a reduction in pressure within the vacuum system might slow the reaction enough to allow a quantitative study of the emittance before the film forms. Therefore, a cold trap (liquid N_2) was added to the system reducing the pressure to about 10^{-6} Torr. Sample 5 and 6 were then prepared and tested; the results appear in Fig. (7) and Fig. (8) respectively. Fig. (7) and Fig. (8) give a graphic account of the formation of this film as indicated by the increase in emittance as time progresses. On sample number 5, measurements were made rapidly at 100C° increments from 800°K to 1300°K at which point the emittance was allowed to equilibrate. The measurements at the previous points were then repeated. The curve in Fig. (7) marked "previous data" is that from Fig. (6) where sample number 4 has been included and is data taken after equilibrium is reached but before the long term aging appreciably roughened the surface. The data points in Fig. (8) are those from measurements on sample number 6 using a technique similar to that on sample number 5, except here, long term aging was applied at high temperatures. A drop in emittance at 1800°K can be noted. Again, the "previous data" curve is from another sample with similar history; in this case, long aging at 1500°C.

Fig. (9) is a composite of smoothed data taken from all samples and shows the three emittance conditions of molybdenum along with the theoretical curve based on resistivity values from Fig. (5). Since the upper curves (C and D) were obtained with several different ribbons and at several different pressures between 10^{-4} and 10^{-6} Torr, they appear to be characteristic of molybdenum in this pressure range, while the lower curve (B) represents polished molybdenum in an ultrahigh vacuum. The data points in Figs. (7) and (8) which were used to establish curve B in Fig. (9) were each obtained in about one minute; hence, the total heating time was approximately five minutes on each sample prior to the stablization at 1300°K to allow the coating to form.

The equilibrium emittance is that of a combination of polished metal and semi-transparent surface film rather than that of the base metal itself. An attempt to determine the composition of this film by x-ray diffraction has tentatively identified it as molybdenum carbide (Mo_2C). In order to discount the possibility that this reaction is unique only to the particular experimental apparatus used, an unheated yet polished portion of sample number 5 was heated in an induction furnace at 10^{-5} Torr with an apparatus quite different from that used in the emittance studies. A film, apparently the same as the other, formed in this system. If this film tended to decompose above a certain temperature, lowering the emittance, while the surface etching and recrystallization tended to raise the emittance, a partial cancellation of effects might be observed. This is one possibility that would lead to the lowering and then raising of the emittance during long aging at 1800°K as can be seen in Fig. (8).

If this phenomenon is only a function of the environmental pressure (and temperature), the result is that any value of total hemispherical emittance lying between curves B and D in Fig. (9) may be obtained depending on the time-temperature-pressure history of the polished sample.

Figure (10) is a display of spectral emittance at 0.65 microns versus temperature. There appears to be considerable scatter in the data depending on temperature and previous heating history. However, when one considers the equation for spectral emittance:

$$\epsilon_\lambda = \exp\left|-2.211 \times 10^4 \left(\frac{T - T_A}{TT_A}\right)\right| \qquad (1)$$

where T is the true temperature in °K and T_A is the brightness temperature, it follows that:

$$\left|\frac{\Delta\epsilon_\lambda}{\epsilon_\lambda}\right| = \frac{2.211 \times 10^4}{T} \left|\frac{\Delta T}{T}\right| \qquad (2)$$

This gives the percentage error in spectral emittance for a given error in temperature. As an example, take a 1 per cent error in temperature at 1500°K. The result is an error of approximately 15 per cent in spectral emittance. Consequently, it is not surprising that, due to the subjective nature of an optical pyrometer measurement as well as small uncertainties in the true temperature, a wide scatter could occur in small sampling as in this case. More conclusive results could probably be realized with repeated measurements. Qualitatively it appears that aging at high temperatures tends to raise, while polishing tends to lower, the spectral emittance at 0.65 microns.

Figure (11) is a plot of the ratio of total hemispherical to total normal emittance versus temperature for the oxidized molybdenum surface and Fig. (12) is a plot of this ratio versus total normal emittance along with the theoretical curve based on the equations of Foote,[4] and Davisson and Weeks[3].

Figures (13) and (14) are graphs of the angular distribution of intensity relative to the normal, plotted against the angle θ and $\cos\theta$, respectively, at 1210°K for the stablized molybdenum surface.

TIME VARIATION OF EMITTANCE

One of the observations recorded in the previous report was a change in both the total and spectral emittance of polished platinum as it was aged at high temperature. This phenomenon is more readily observed if the surface of the sample is initially very highly specular. These results show a general increase in emittance with time which is caused, in part, by thermal etching and by the recrystallization of the bulk metal. Work on the time variation of emittance has been continued in order to see if there is any regular relationship between the rate of change and the temperature and also between the final value of emittance after aging and the temperature. It is also desired to observed the effect of the initial surface condition on the variation of emittance with time. The research was continued with platinum for correlation with the results of the previous investigation.

The parameters to be measured as a function of time were the total hemispherical emittance, the normal spectral emittance at 0.65 microns, the electrical resistivity, and the surface microstructure. The electrical resistivity was recorded in addition to the thermocouple voltage as a check on the constancy of the temperature during the aging period. The surface microstructure was observed during the course of the aging process to help explain the reasons underlying the emittance changes.

To determine these parameters, it was necessary to have an accurate knowledge of the current supplied to the ribbon, the potential drop across a uniform temperature region at the center, the absolute temperature of this region, the brightness temperature at 0.65 microns, the ambient temperature of the chamber housing the ribbon, and the geometry of the radiating surface. All of this information was obtained regularly at the end of a series of predetermined time intervals.

The total hemispherical emittance, electrical resistivity, and the spectral emittance at 0.65 microns were determined by techniques described in the previous report. The electrically heated ribbon was suspended in a vacuum of 10^{-4} to 10^{-5} mm of Hg. The true temperature was monitored by a pt vs pt-rh thermocouple welded at the midpoint of the ribbon. The potential drop was measured by two voltage leads which were welded 2.5 cm from either side of the thermocouple. The brightness temperature was measured by a micro optical pyrometer which viewed the center portion of the ribbon through a quartz window in the vacuum chamber. The necessary rigidity and the motion required for optical alignment was accomplished by mounting both the vacuum chamber and photographic system on an optical bench with standard carriages. The chamber was isolated from the pumping system by means of a 1 - 1/2 inch flexible metal hose thus eliminating the vibration from the roughing pump. The photo-optical system consisted of a 16 mm cine Kodak motion picture camera modified by a system of a.c. solenoids connected to a mechanical shuttering device. The repetition rate could be varied from

one frame per second to one frame per 30 seconds. In this manner, not only a photomicrographic record of the sample was obtained at high temperature, but also an accurate time base for the measurements was established. The camera was coupled to a telemicroscope which magnified the surface by a factor of 40 to 1. The telemicroscope had an objective lens with a focal length of 48 mm making it necessary to place the sample within 45 mm from the end of the microscope. This meant that the ribbon had to be very close to the viewing port of the vacuum chamber. The entire vacuum chamber was water cooled to carry off the heat and a fan was used to cool the window of the viewing port.

In order to take photomicrographs having good definition, it was necessary to illuminate and view the ribbon surface normally. This was accomplished with a glass plate which reflected a parallel light beam to the sample, which in turn reflected the incident light back from the surface to the telemicroscope.

In the final set up shown in Fig. (15), the power was supplied to the specimen from a voltage regulated a.c. source; the potential drop between the voltage probes was measured by an a.c. voltmeter with an accuracy of 0.25 per cent of full scale. The current was monitored by an a.c. ammeter of the same accuracy in conjunction with a 10 - 1 step down current transformer. The emf from the thermocouple was measured with a potentiometer, and the brightness temperature was measured by a micro optical pyrometer.

The power to the ribbon and the time delay camera was turned on simultaneously and when the specimen reached the desired thermocouple temperature, the values of the other parameters were recorded. In order to obtain a reasonable idea of the emittance changes with time, the measurements were extended for a period of 100 minutes or longer at a given temperature. During this time, data were recorded every 15 minutes. The emf of the thermocouple was held constant by controlling the power to the ribbon. Photomicrographs were taken at the rate of 1 frame every 30 seconds. The accuracy of the electrical power dissipation determination is $\pm$ 1/2 per cent, since it is derived from current and voltage measurements, each with a stated accuracy of $\pm$ 1/4 per cent. The absolute value of the electrical resistivity may have a greater uncertainty due to the difficulty of determining the cross sectional area as mentioned in the previous section. Even though the thermocouple voltage could be read quite accurately with the potentiometer, it is felt that systematic errors in the thermocouple circuit of this system have increased the temperature error to $\pm$ 1 per cent.

Sample number 1 was installed in the as-received condition without further surface preparation other than cleaning by immersion in acetone and rinsing in distilled water. The measurements were made with preliminary instrumentation since the precision instruments which have the above accuracy were unavailable at that time. Thus the data received from these observations are less reliable than those taken on subsequent ribbons. The measurements were made at 1310°K with a total time of 285 minutes. As seen in Fig. (16), the emittance seemed to attain equilibrium in approximately 90 minutes, changing initially from 0.163 to a final value of 0.171 for the total hemispherical emittance and from 0.35 to 0.46 for the spectral emittance.

These were increases of about 5 and 30 per cent respectively. The precision of the instrumentation was not great enough to state definitely that this increase was a real effect. A small error in temperature could easily cause errors of this magnitude with relatively larger errors in the spectral emittance than in the total emittance. A 1 per cent error in the temperature at 1310°K would cause a 4 per cent error in total emittance and a 17 per cent error in spectral emittance provided the measurement of electrical power dissipation and brightness temperature was accurate.

Sample number 2, which had previously been held at 1000°K for several hours with a platinum black surface for calibration purposes, was cleaned and repolished by hand using aluminum oxide and crocus cloth giving it a highly specular surface. The measurements were made with more precision than those of sample number 1. Sample number 2 was run at 1310°K on three consecutive days, adding up to a total time at temperature of 750 minutes. The results are shown in Fig. (16). The initially rapid rise in the total emittance was not observed for this specimen. For the first 200 minutes, there was little change in the total emittance, but subsequent to this time, it experienced an increase of 1 per cent per hour finally changing by 10 per cent over the entire measurement period. The total increase in the spectral emittance during the entire measurement is 22 per cent. The reason for the difference in the initial spectral emittance of the two specimens is not known. The film record in Fig. (17) from sample number 2 at elevated temperature gives some indication of why the emittance characteristics change as they do. Starting from a polished state, this sample gained a mottled appearance which by two hundred minutes was well defined into areas of higher and lower emittance. By four hundred minutes, the crystals were well defined in the high and low emittance areas taking the form of individual crystal faces. By the end of the measurement period, the crystal interfaces were very well defined. While the definition of these interfaces increased with time, no definite change in the size of the crystal was observed. Apparently the thermal etching proceeds more rapidly than recrystallization at this temperature.

Under higher magnification of the cold ribbon, as seen in Fig. (18), the high emittance crystal faces are resolved into areas displaying many little pits. These pits are concentrated in particular crystal surfaces such that they define its area quite well. In addition, there is a low concentration of pits in all the crystal faces. A rough estimation indicates that the areas of the pits accounts for about 10 per cent of the total surface area of the ribbon. Since this phenomenon was not observed on the photomicrographs of sample number 1 or any of the previous strips, it is felt that it may be due to the previous chemical, mechanical, or thermal treatment of this ribbon.

Sample number 3 had no previous high temperature history and was treated only by polishing with the rotating drum attachment described in the last section. The resultant surface was highly specular although grooves can be seen under magnification (see Fig. (19)). These grooves which were probably caused by the initial rolling process and brought out by polishing did not appear to be sufficiently deep or numerous to appreciably effect the emittance.

The ribbon was held at 1200°K, 1350°K, 1500°K, and 1600°K, respectively, until the emittance at each temperature appeared constant. Then the temperature was reduced to 1500°K, 1350°K and 1200°K in descending order allowing the emittance to stablize at each temperature. A complete cycle required 900 minutes. Figure (20) is a plot of the total hemispherical emittance versus time for these measurements. Data for the spectral emittance also were recorded but the results were not interpretable and have not been included. The measured emittance increases to its equilibrium value in approximately the same time at each temperature. When the temperature is decreased, the emittance decreases to an equilibrium value which is slightly higher than the equilibrium value obtained during the increasing temperature steps. The rather rapid changes in measured emittance with time appeared to be more characteristic of an instrumental error than a real effect. The source of these changes was then investigated further. However, the increase in the equilibrium value at a given temperature, between the increasing and decreasing steps, is assumed to be real, but the amount of increase which was in order of 1 per cent in the time intervals measured was still too small to be determined very accurately.

Sample numbers 4 and 5 were from the same lot of platinum as sample number 3. The pre-treatment and polishing techniques were the same, except for the fact that the final cleaning was accomplished with an ultrasonic cleaner. The microgrooves present in sample No. 3 did not appear in samples 4 and 5 and the surfaces were visually very specular in both cases.

Measurements of the total hemispherical emittance versus time were taken on sample number 4 at 1500°K, 1700°K, and 1682°K for a total period of 1200 minutes. The results of these measurements are recorded in Figure (21).

In the previous runs, the thermocouple was attached to solid kovar electrical feed-through terminals within the vacuum chamber, while the cold junction and measuring circuit were attached to the outside extension of these terminals. It was assumed that the terminals were placed so that there was no temperature differential between them or any temperature gradients along the kovar rods. However, the behavior of the data indicated that this might not have been a safe assumption. For samples numbers 4 and 5, continuous thermocouple wires were installed from the high temperature junction to the reference junction outside the vacuum system and from there to the measuring circuit.

Both the platinum and platinum 10% rhodium wire were fed through hollow kovar terminals in the base of the system: the terminals were mechanically crimped and then soldered at one end to form a vacuum tight seal. The thermocouple wires were insulated from the rest of the feed-through so that no thermal junctions could be formed that would be sources of undesired voltages.

It can be seen in Fig. (21) that there was essentially no change of emittance with time at 1500°K over a period of ~ 490 minutes. The small

decrease in emittance during the first hour was probably due to a thermal polishing effect on the micro scratches caused by the polishing wheel. At the end of this period the temperature was increased to 1700°K for 180 minutes. At this temperature, there was an observable increase in emittance over the entire time period. This was in the order of 1% total, or 0.33% per hour. The temperature was then reduced to 1500°K for 60 minutes. This showed a total increase of ≈ 0.5%.

The next day, the ribbon was set at a temperature of 1660°K for 422 minutes. The measurements indicates an increase in total hemispherical emittance of ≈ 0.3% per hour. Photomicrographs showing the initial surface, the final polished surface, and post run surface are included in Fig. (22).

Sample No. 5 was reserved for one last measurement of total hemispherical emittance versus temperature from highly polished platinum over a temperature range of 773° to 1773°K. These data were taken rapidly enough so that there was essentially no change in the surface condition of the ribbon. This measurement extended the high temperature range of the previous NPDL data by 300°K, and also compares the results with those published in the first report for a freshly rolled highly specular but unpolished strip of platinum. These data are included in Fig. (23) and shows a correlation to within ±5%.

The photographic records for samples number 2, 3, 4, and 5 shown in Figures (17), (18), (19), and (22) indicate that the crystals apparently did not change in size with time at these temperatures, although their very faint outlines became more defined as time progressed. The depth of the interfaces after a complete run is not known and the width can only be estimated to be in the order of 1 micron while the distance across the crystal face vary between 25 and 300 microns. Thus the only mechanical effect other than the pits noted on sample number 2 which could change the emittance would be changes in the size and depth of the crystal interfaces. The orientation of all the crystal faces was observed by reflection to be essentially parallel to the macroscopic surface of the ribbon.

DISCUSSION OF RESULTS

The equilibrium values of the total hemispherical emittance of the six strips of molybdenum tested were about 60 per cent higher than that predicted by the theoretical equation of Davisson and Weeks[3] using the measured values of the electrical resistivity. The value of emittance was reproducible among the six strips even though the surface treatment was somewhat different in the six cases. The appearance of a slight coating on the surface is the cause of the increase in emittance over the theoretical value. This may be characteristic of molybdenum in this pressure range of from 10^{-4} to 10^{-6} or it may be due to outgassing of the measurement chamber or to untrapped vapor from the oil diffusion pump. The emittance characteristics of the coating must not be too pressure sensitive because the pressure in the chamber was not identical in the four different runs. Measurements made rapidly when the strip was first heated have produced values of emittance which are more characteristic of the polished surface and are more nearly in agreement with the theoretical equation. It is noted in the comparison

of Figures (9) and (23) that the deviations between the theoretical curve and the measured values are similar for molybdenum and platinum.

The results on the variation of emittance with time have succeeded in showing that there is a slight increase due to thermal etching or recrystallization, but the exact amount of this enhancement is very small and was not determined accurately enough to state quantitatively at this time. The principle difficulty lies in an uncertainty of about ± 1 per cent in the temperature determination in the chamber used for this study. This leads to an error of ± 4 per cent in the total emittance and from ± 20 per cent to ± 10 per cent in the spectral emittance over this temperature range. Over the time intervals measured of a few hours, the observed changes are no larger than these stated accuracies and cannot be precisely determined from the present data. Further refinements can be made in the system to permit more accurate values, but in most cases of practical interest, such small temporal changes in a quantity which varies considerably from one surface to another on the same type of material are probably not too significant.

The concentration of effort will now be directed toward meeting the other goals of this project, namely the total hemispherical and the total normal emittance of the protective coatings for the refractory metals as well as the total emittances of their polished substrates.

REFERENCES

1. G. L. Abbott, N. J. Alvares, and W. J. Parker, "Total Normal and Total Hemispherical Emittance of Polished Metals," U. S. Naval Radiological Defense Laboratory, WADD-TR-61-94, July, 1961.

2. A. Goldsmith and T. E. Waterman, "Thermophysical Properties of Solid Materials," Armour Research Foundation, WADC TR 58-476, Oct. 1958.

3. C. Davisson and J. R. Weeks, J. Opt. Soc. Amer. 8, 581 (1924).

4. P. D. Foote, Bull. Nat. Bur. Stand. 11, 607 (1914/1915).

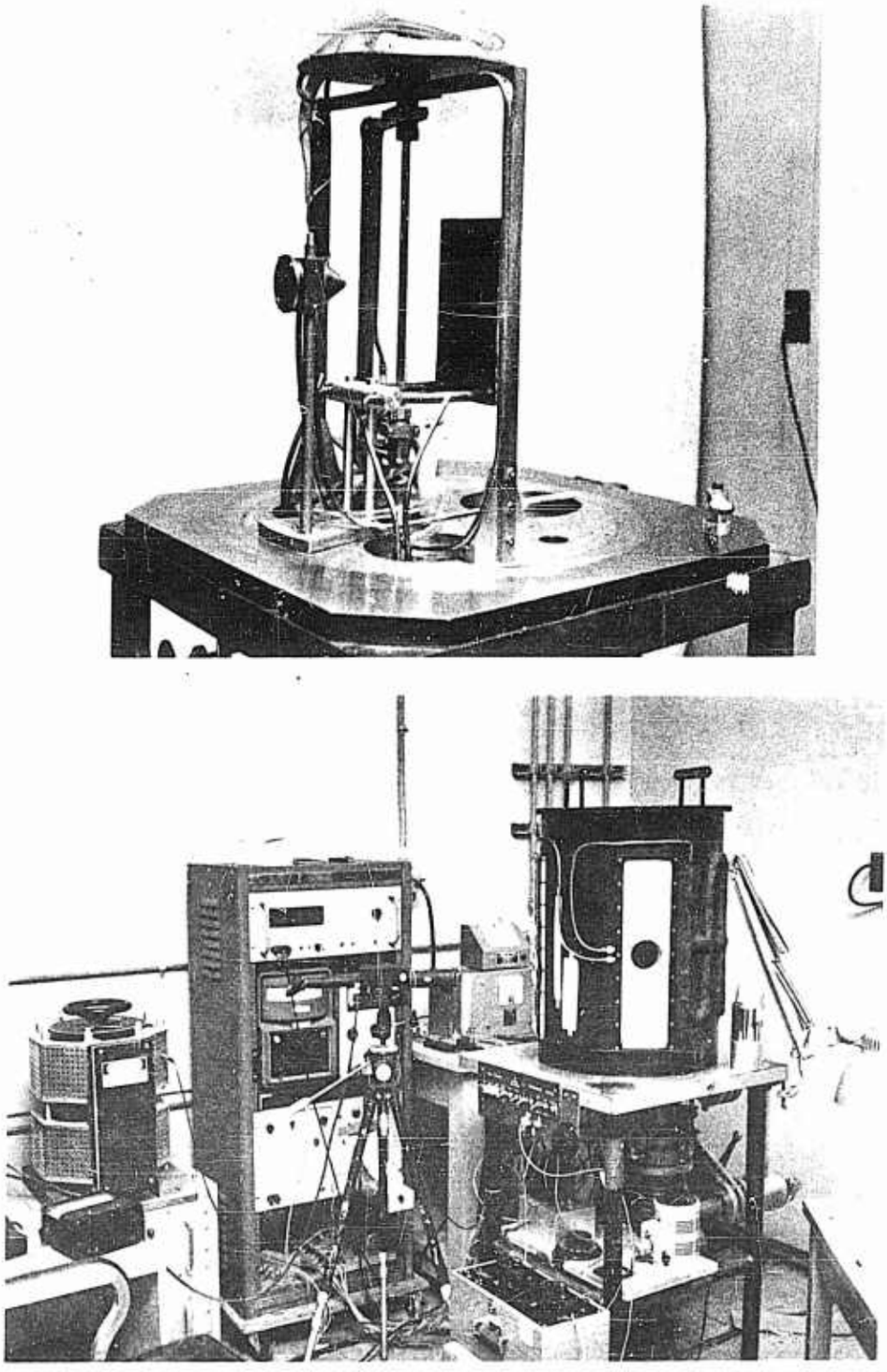


Fig. 1 High Temperature Experimental Apparatus

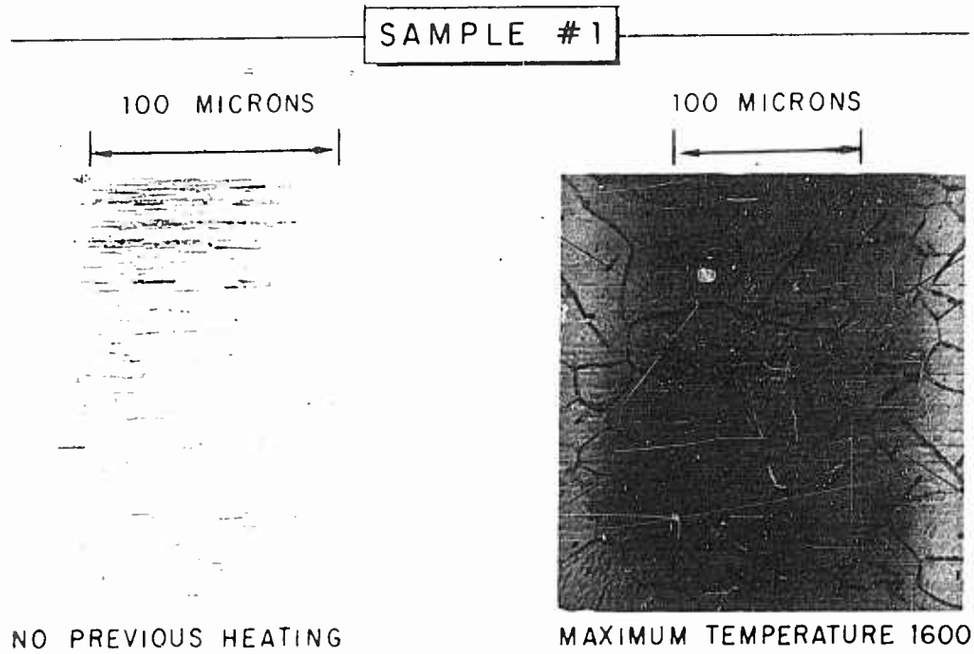


Fig. 2 Photomicrographs of Molybdenum Samples 1 and 2

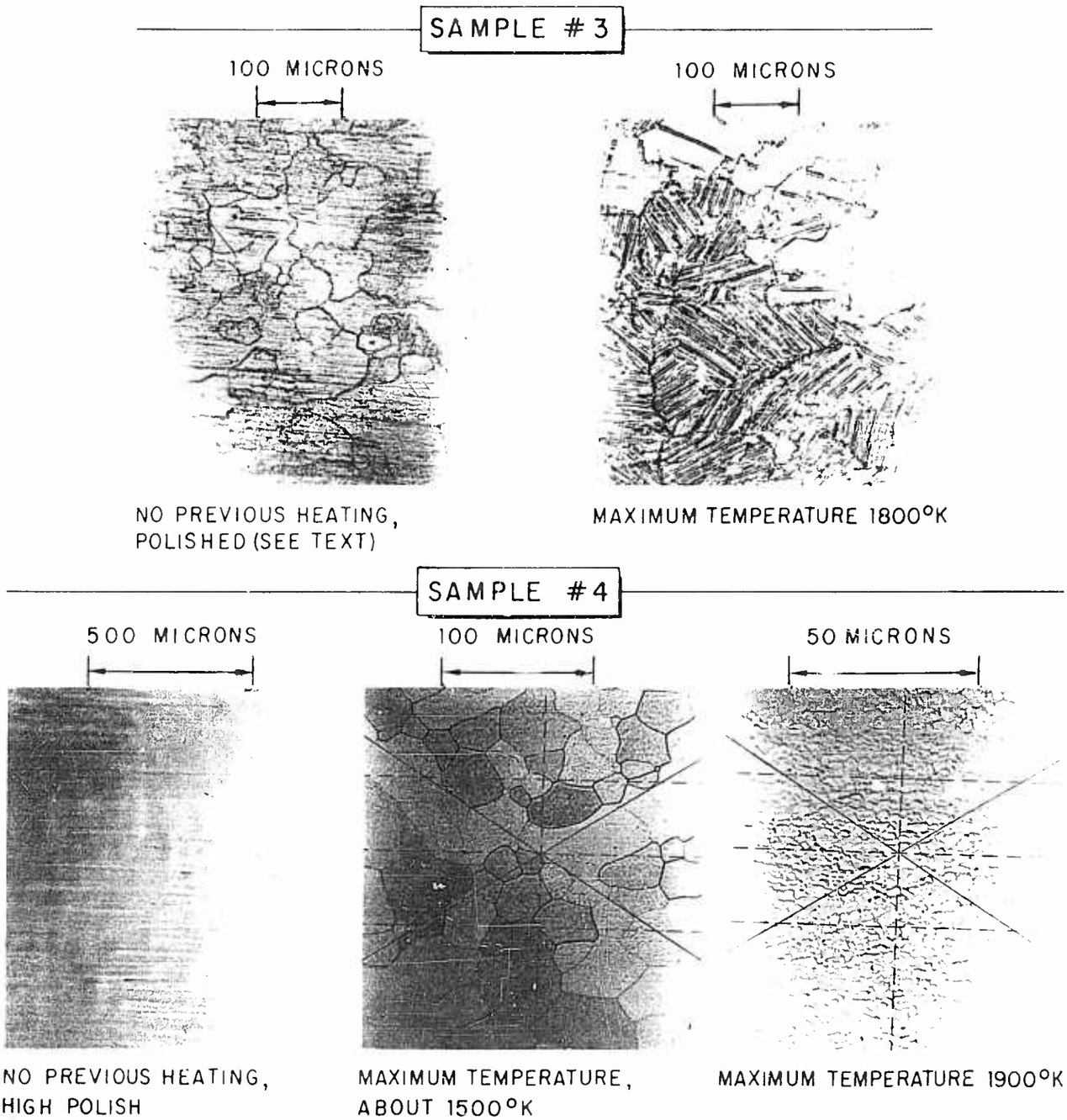


Fig. 3 Photomicrographs of Molybdenum Samples 3 and 4

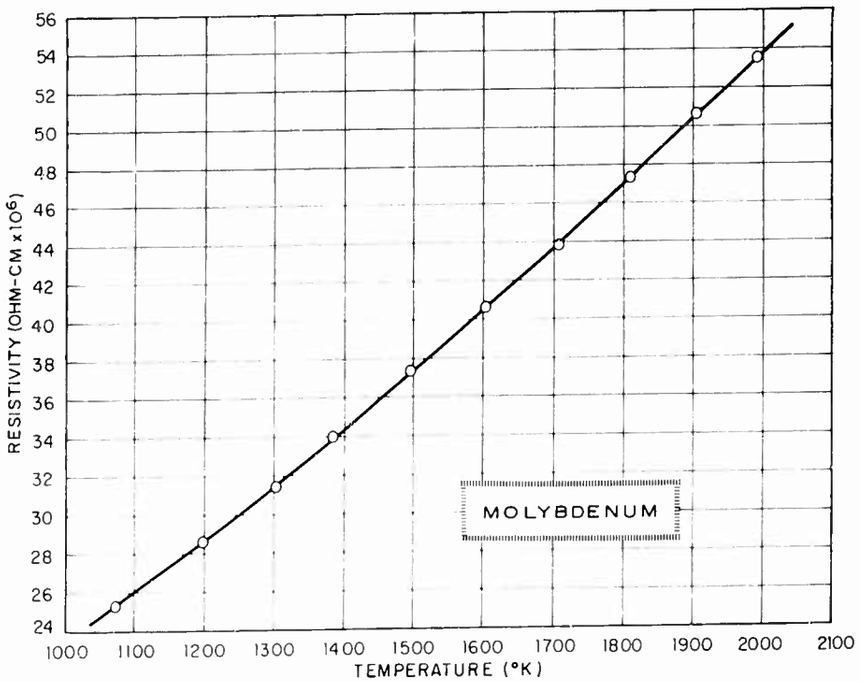


Fig. 4 Electrical Resistivity of Molybdenum, Sample 1

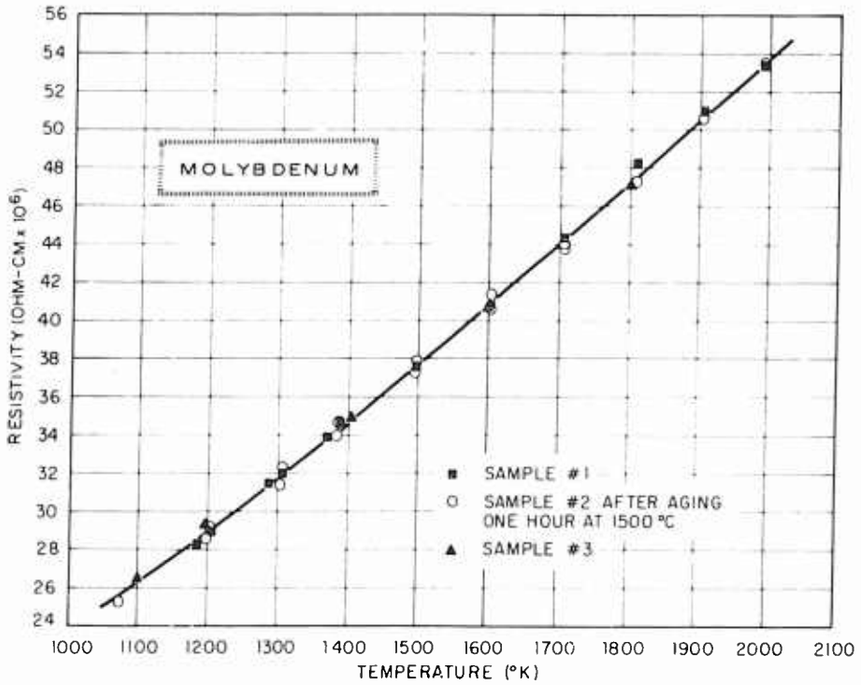


Fig. 5 Electrical Resistivity of Molybdenum, Samples 1, 2 and 3

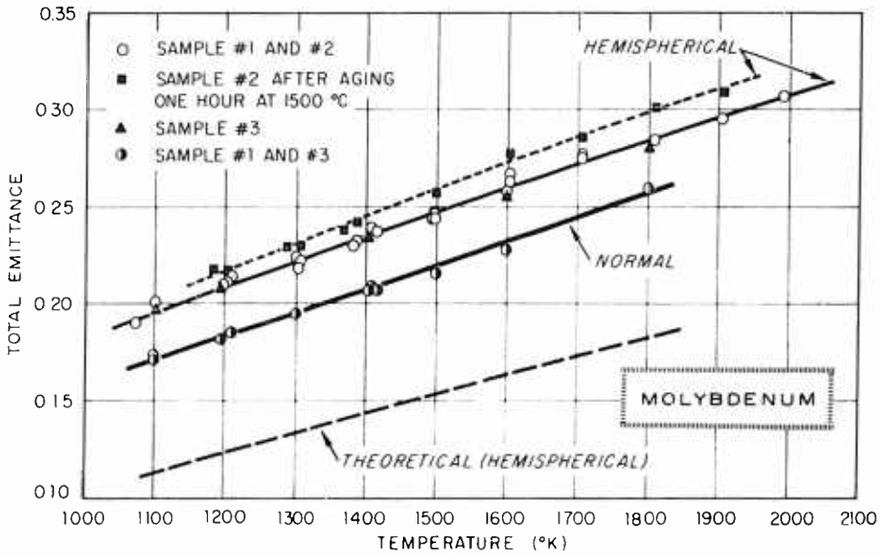


Fig. 6 Total Emittance of Molybdenum

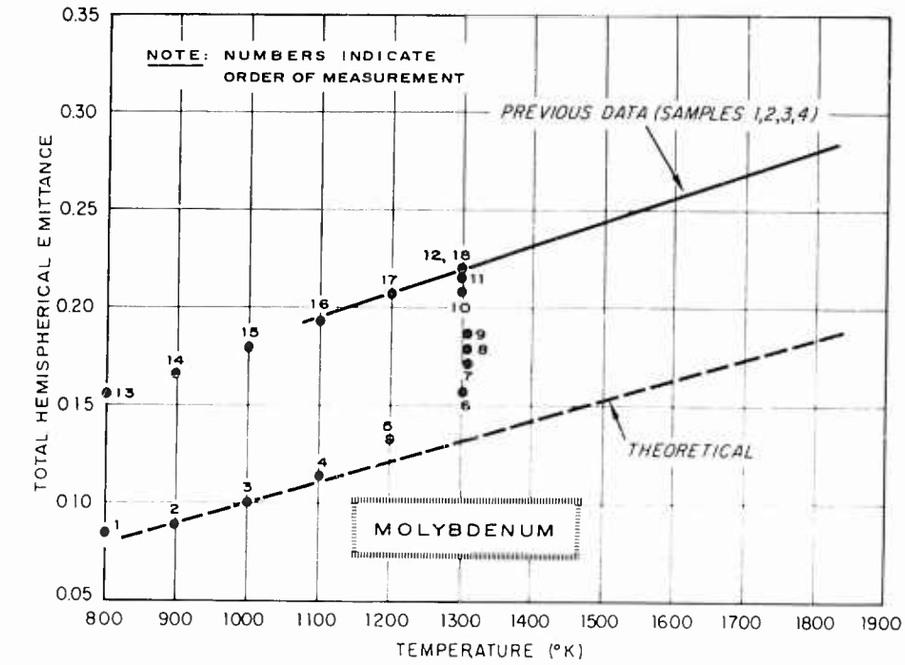


Fig. 7 Total Hemispherical Emittance of Molybdenum, Sample 5

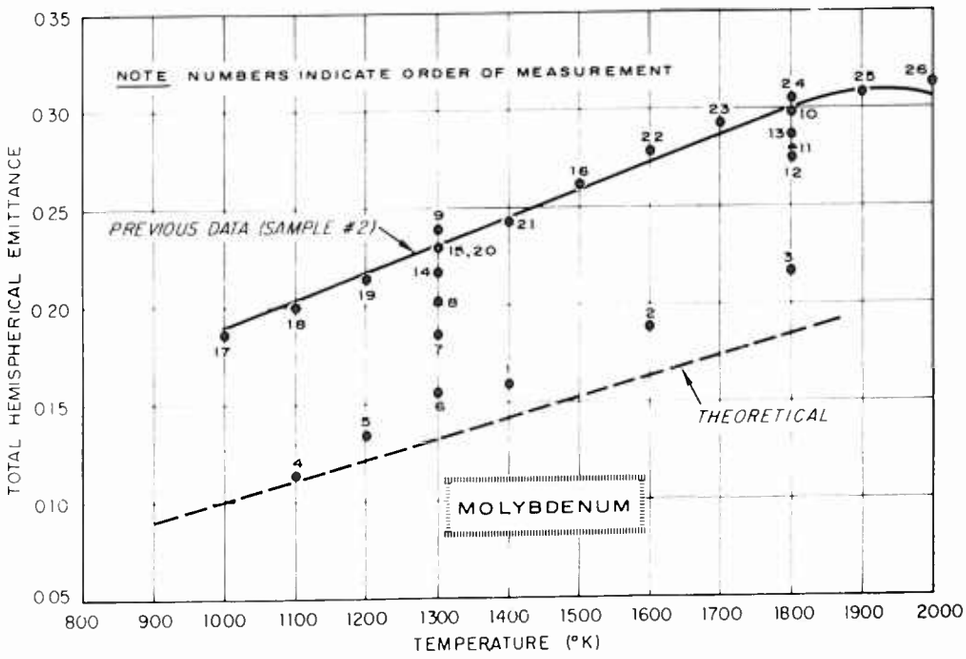


Fig. 8 Total Hemispherical Emittance of Molybdenum, Sample 6

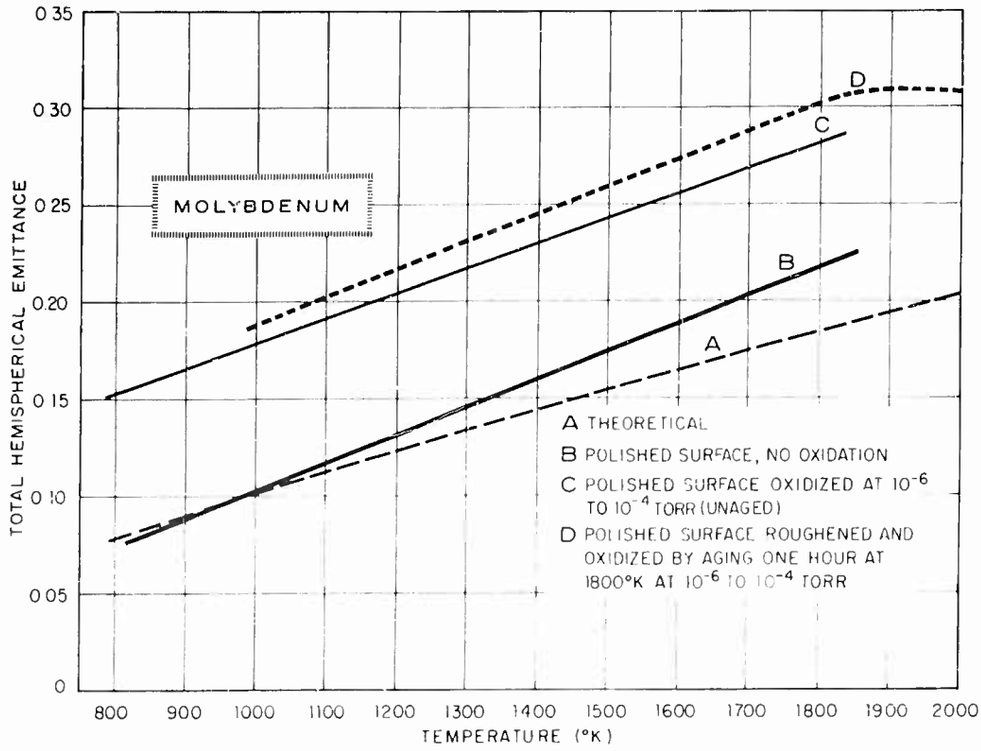


Fig. 9 Total Hemispherical Emittance of Molybdenum (composite, all samples)

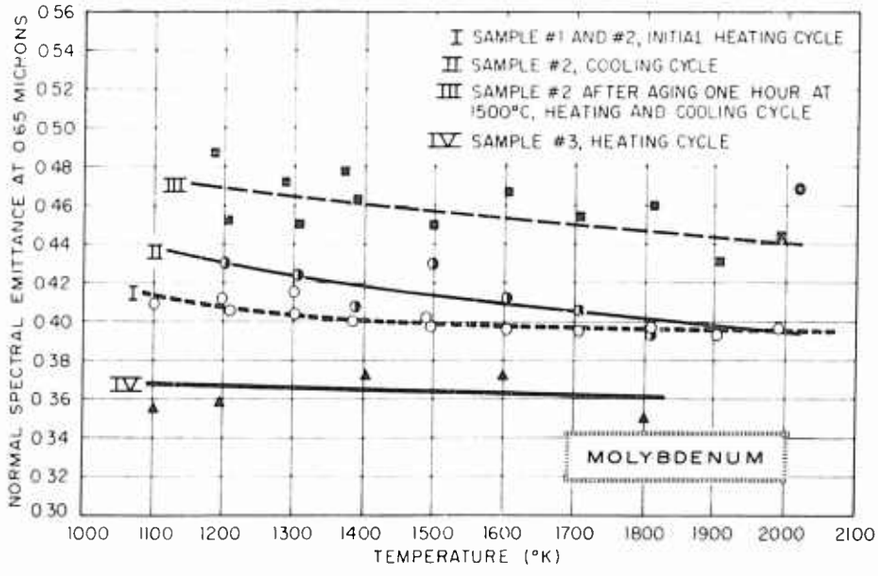


Fig. 10 Spectral Emittance of Molybdenum

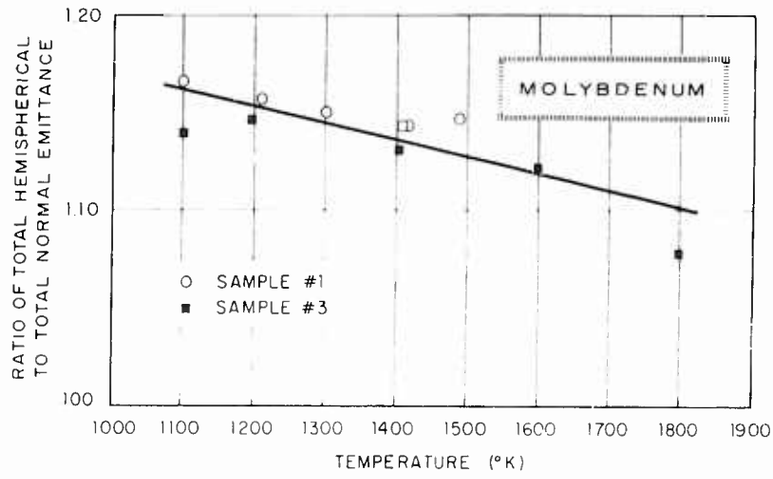


Fig. 11 Ratio of Total Hemispherical to Total Normal Emittance Versus Temperature

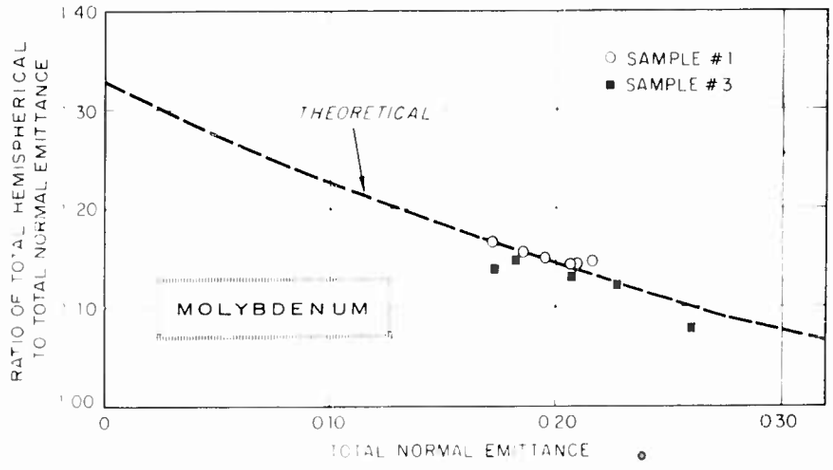


Fig. 12 Ratio of Total Hemispherical to Total Normal Emittance Versus Total Normal Emittance

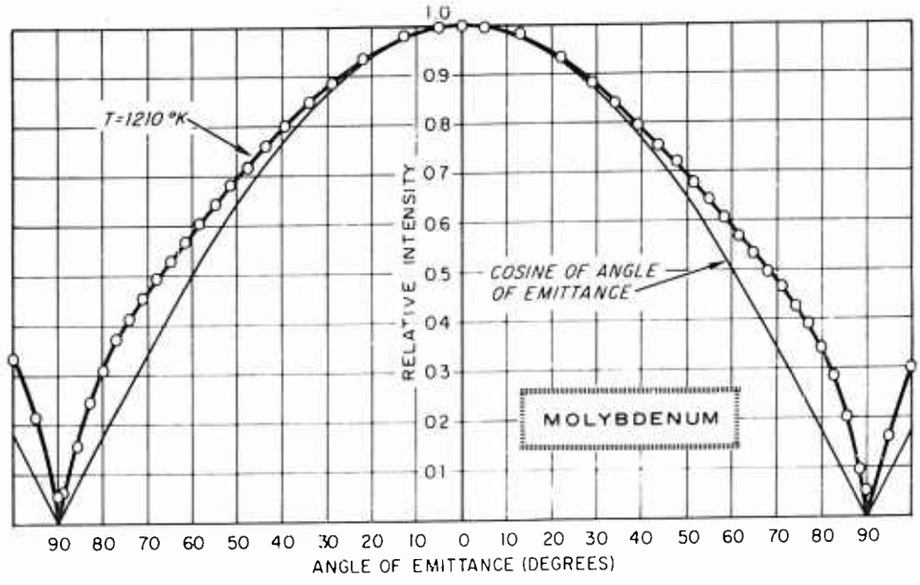


Fig. 13 Angular Distribution of Intensity Versus Angle

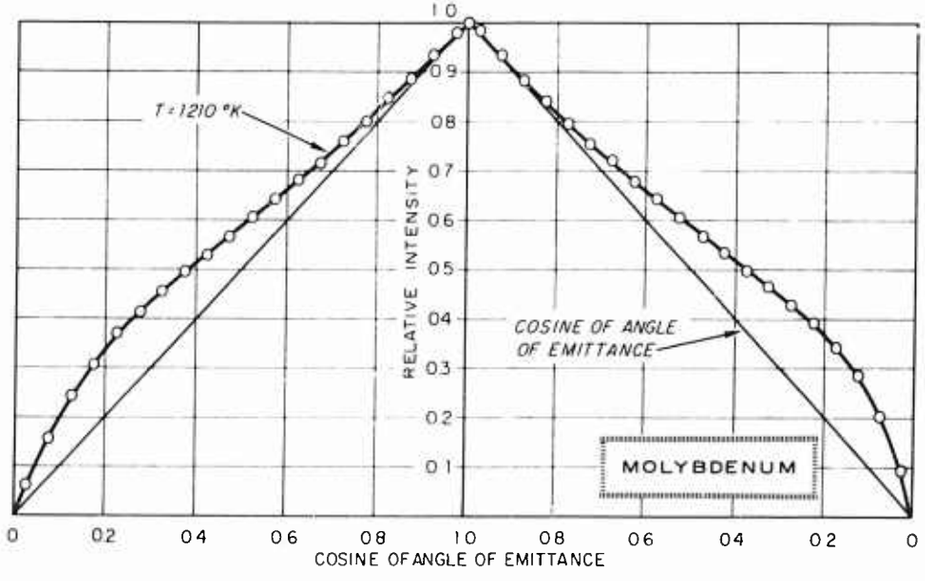
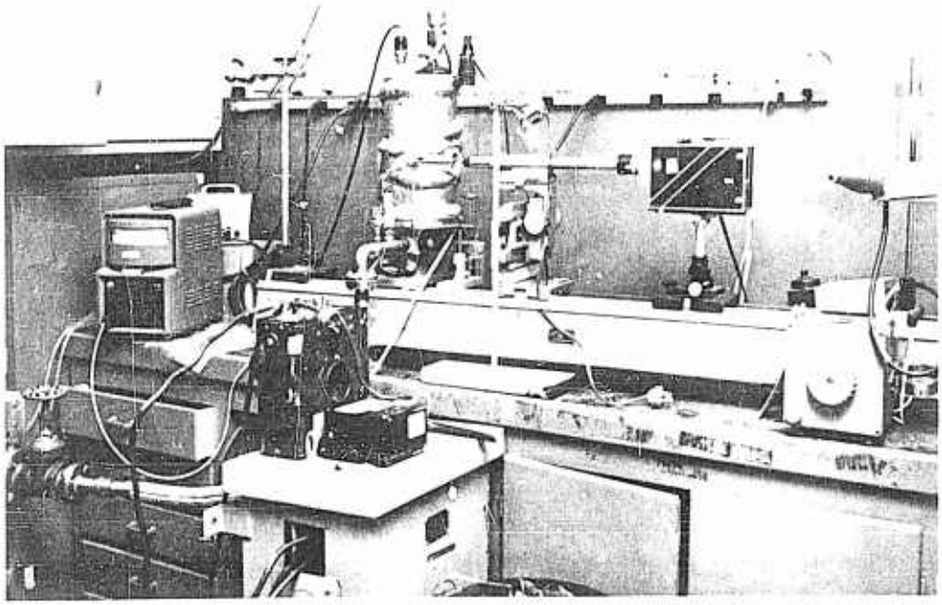
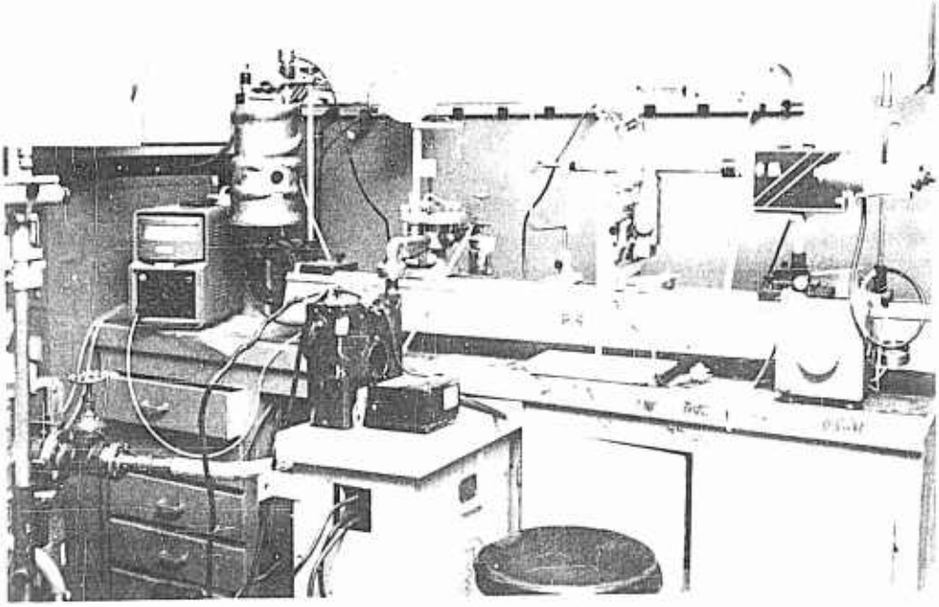


Fig. 14 Angular Distribution of Intensity Versus the Cosine of the Angle





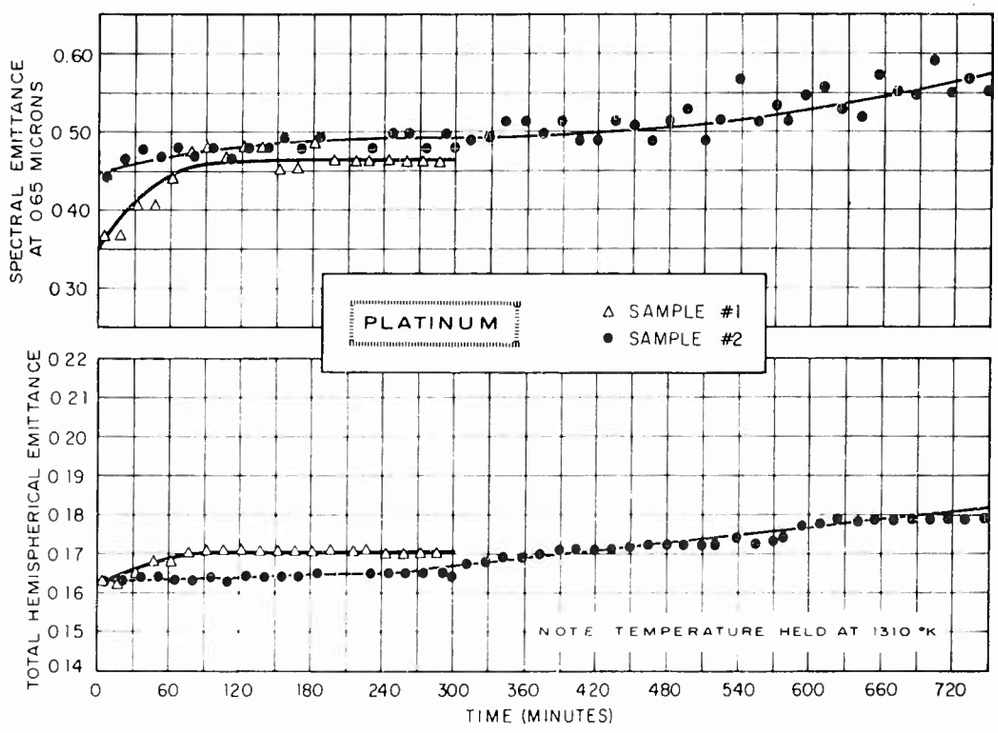


Fig. 16 Emittance of Platinum Versus Time, Samples 1 and 2

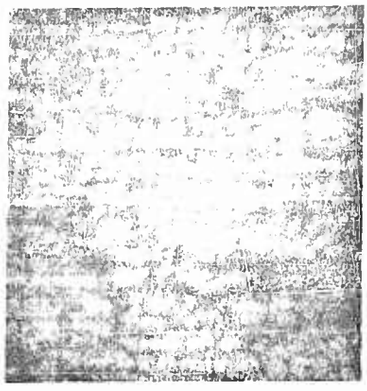


PLATE a, 0 MIN

PLATE b, 266 MIN

PLATE c, 516 MIN

PLATE d, 573 MIN

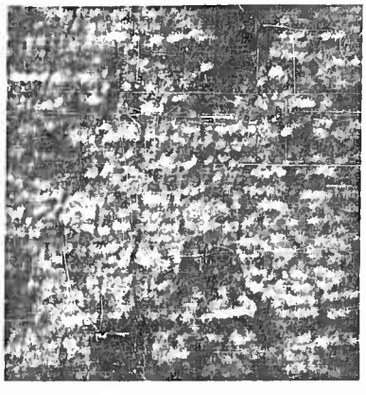


PLATE e, 666 MIN

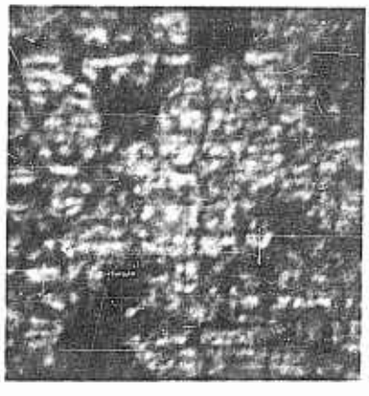


PLATE f, 740 MIN

PLATE g (POST RUN)

PLATE h (POST RUN)

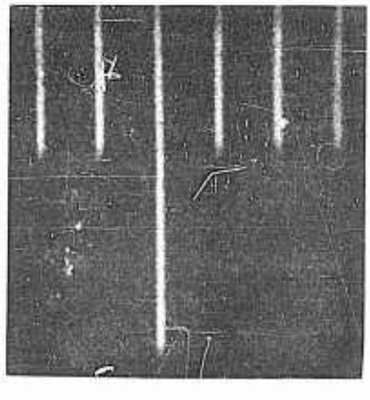


PLATE i (CALIBRATION)

Fig. 17. [illegible] photography of Platinum sample 2 at 1510°K showing the [illegible] surface microstructure from the initially polished state (Plate a) to the final state at 740 min. (Plate f). Plate [illegible] radiation emitted directly from the surface, at 1410°K; the same area photographed by reflected light is shown in Plate h. Plate i is a photograph of the reticle used to calibrate the system [illegible] equals 100 microns.

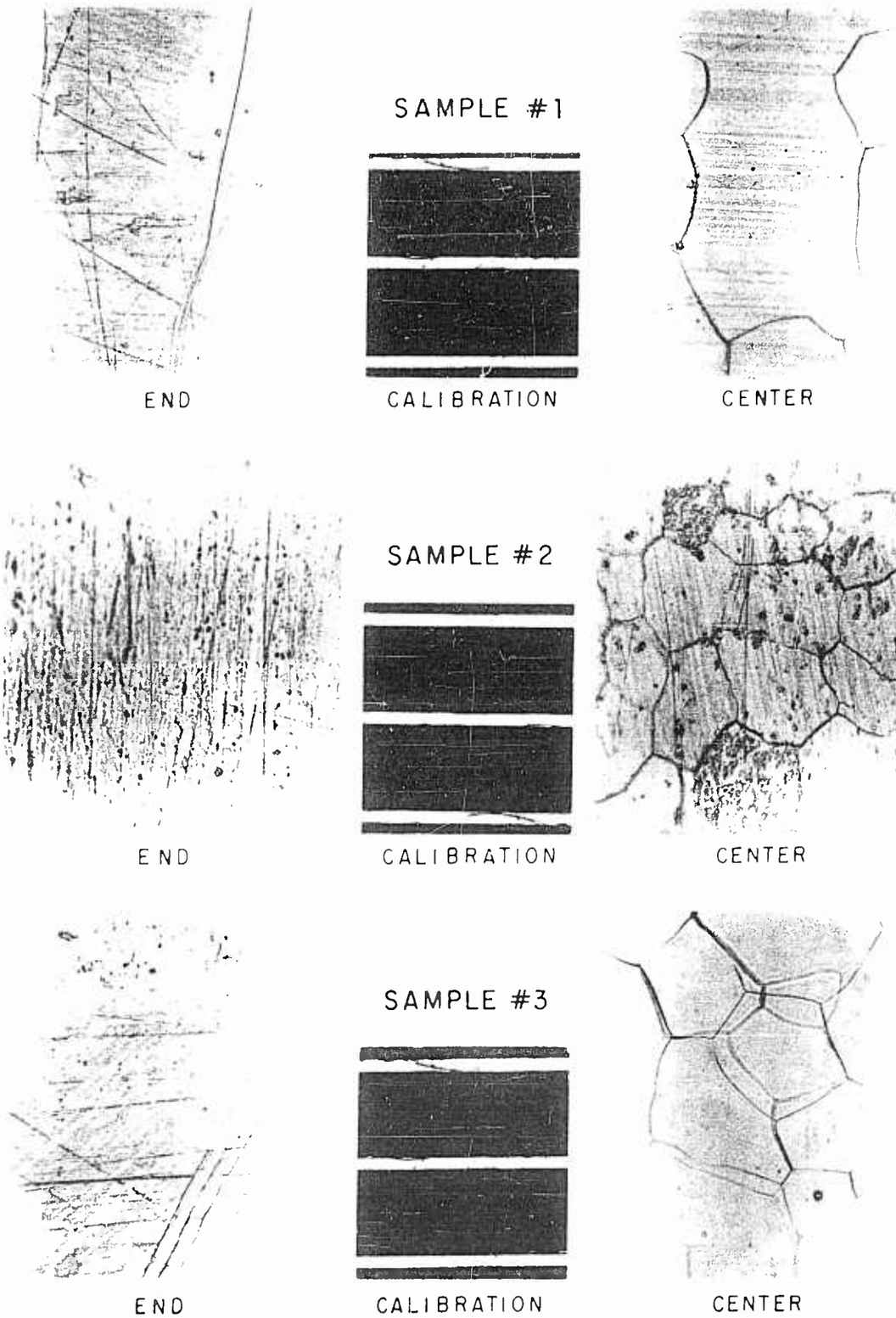


Fig. 16 Photomicrographs of Platinum samples 1, 2 and 3 showing the difference in the surface microstructure between the unheated ends and the uniform temperature regions of each sample. The distance between the divisions of the calibrations equals 100 microns.

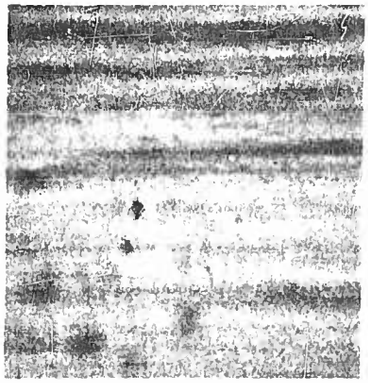


PLATE a, 0 MIN

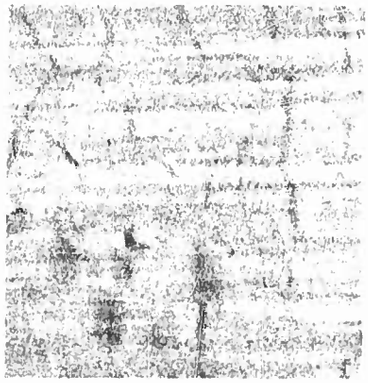


PLATE b, 207 MIN

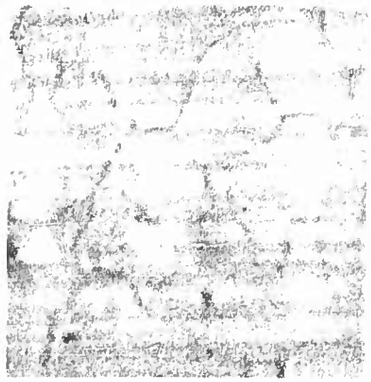


PLATE c, 443 MIN

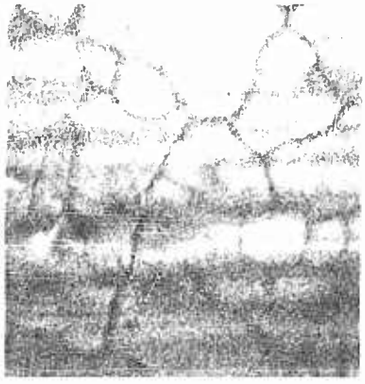


PLATE d, 500 MIN

PLATE e, 569 MIN

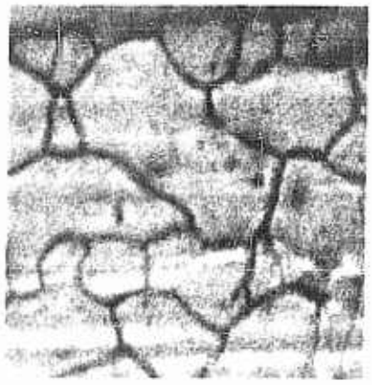


PLATE f, 633 MIN

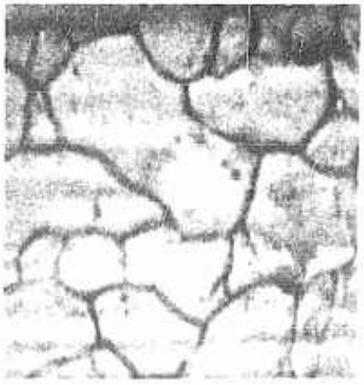


PLATE g, 711 MIN

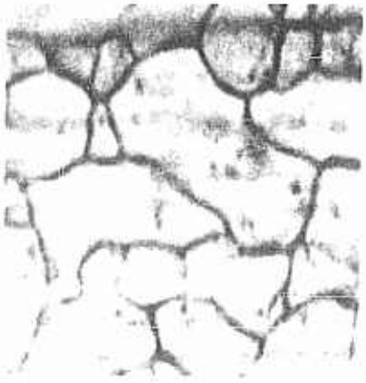


PLATE h, 875 MIN

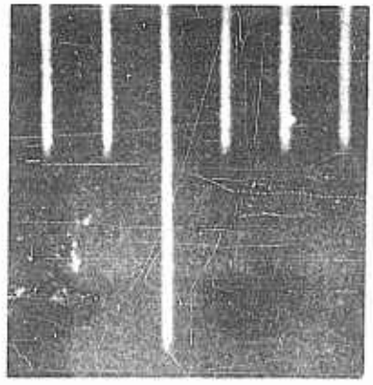


PLATE i (CALIBRATION)

Fig. 1. Time lapse photographs of platinum sample 7 showing the change of the surface microstructure from the initially polished state (Plate a) to the final state at 900 min. (Plate h). Plate i is a photograph of the reticle used to calibrate the system, where 1 division equals 100 microns.

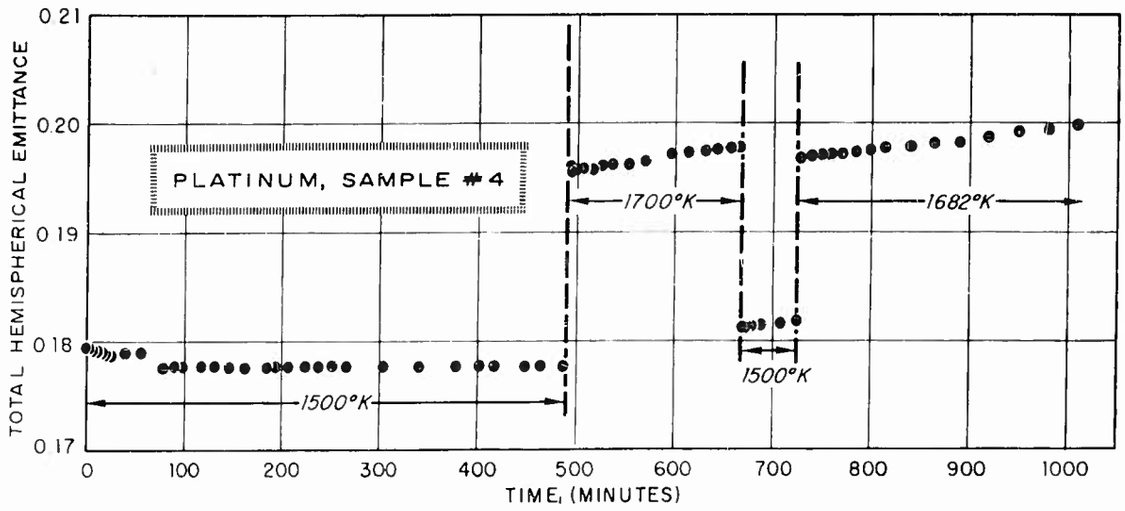


Fig. 20 Emittance of Platinum Versus Time, Sample No. 3

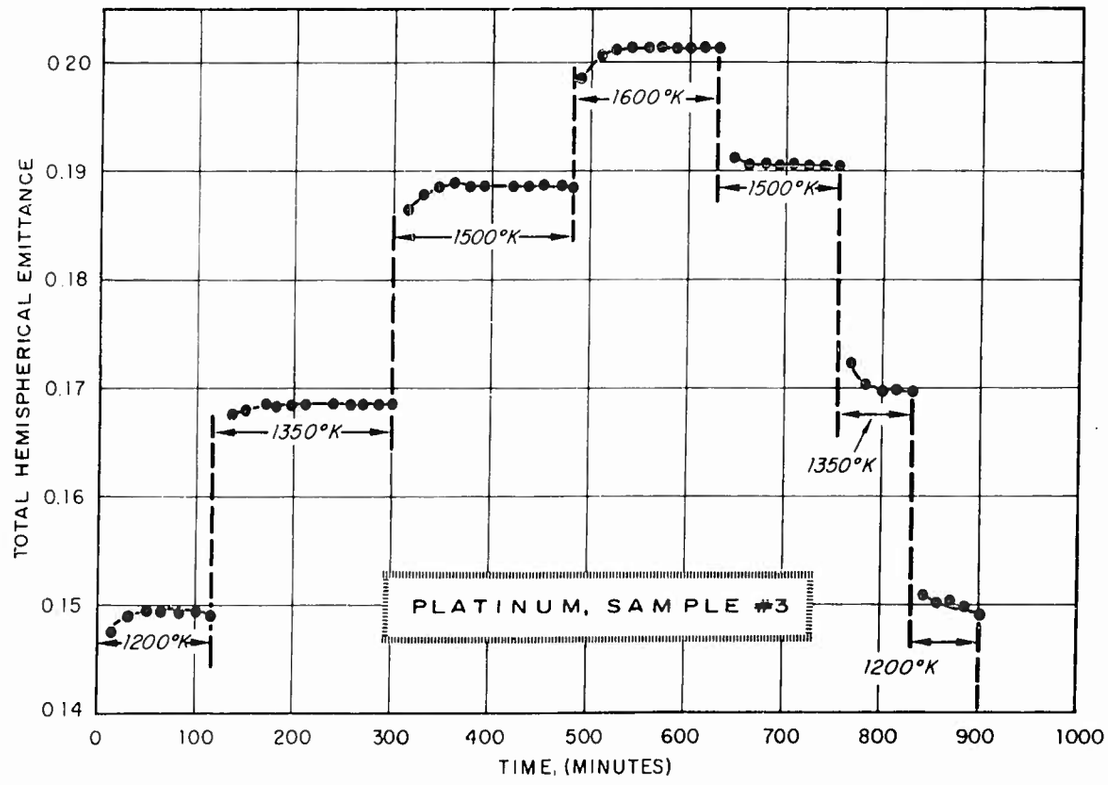


Fig. 21 Emittance of Platinum Versus Time, Sample No. 4

PLATE a

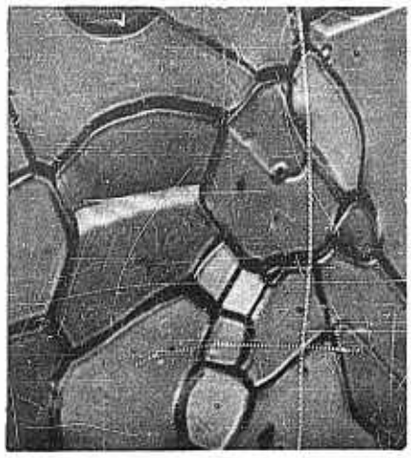


PLATE b

PLATE c

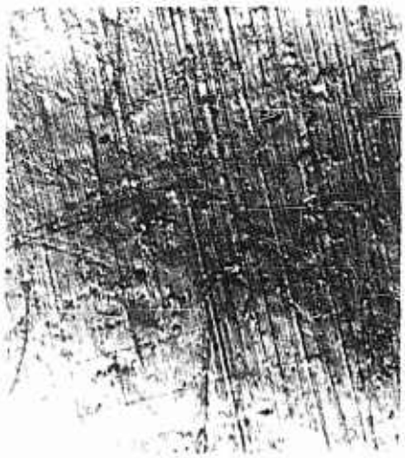


PLATE d

PLATE e

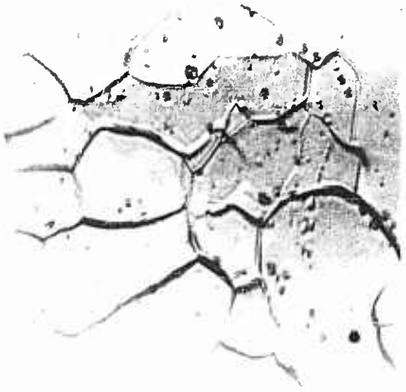


PLATE f

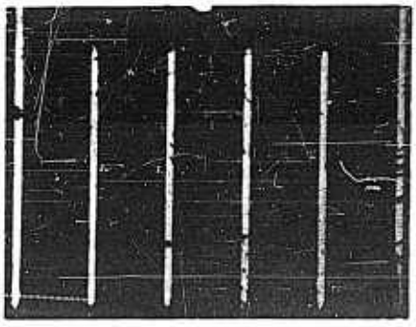


PLATE g

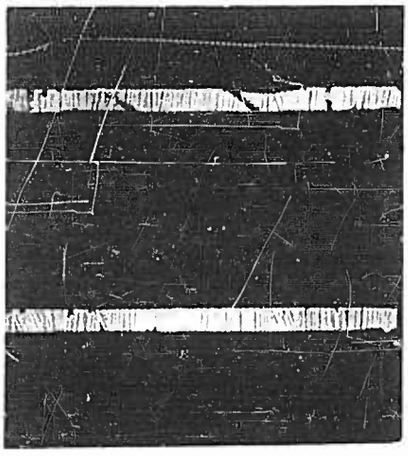


PLATE h

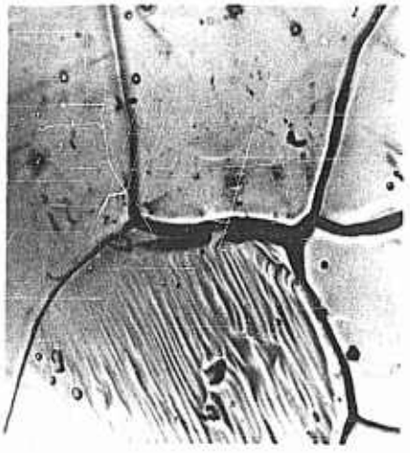


PLATE i

Fig. 22 Photomicrographs of Platinum Samples 5 and 6, with plates a, b, and c chronologically showing the "as received," "polished," and "post run" condition of Sample No. 4 and plates d, e, and f show the "as received," "polished" and "post run" conditions for Sample No. 5. Plate "g" is the calibration for plates a through f where the distance between any two adjacent lines equals 100 microns. Plate h is the calibration for plate i which shows a section of Sample No. 5 at almost three times the magnification of the other photomicrographs.

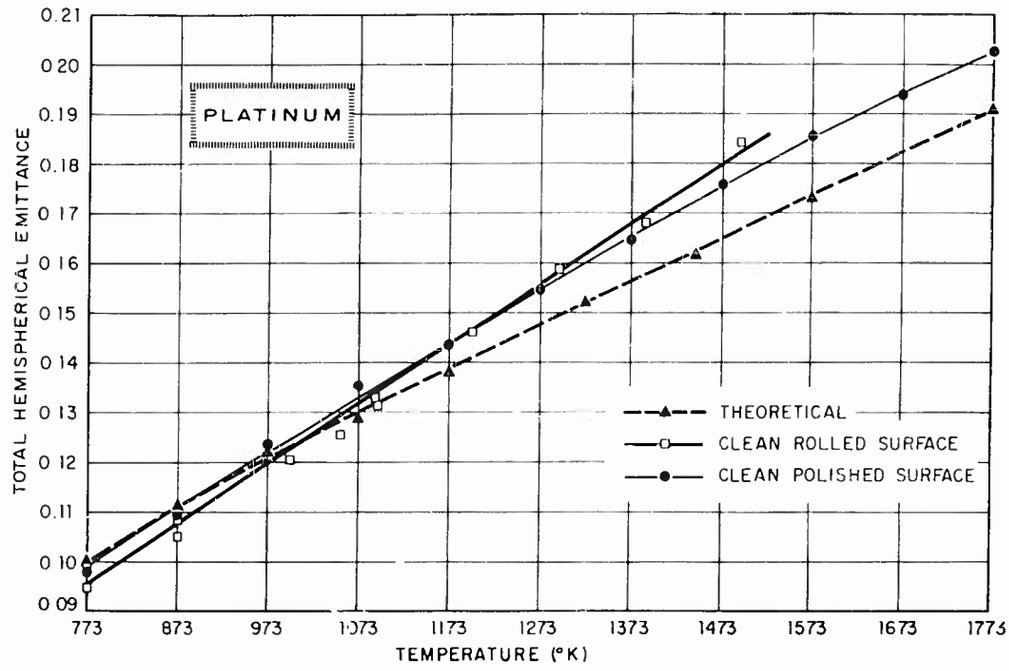


Fig. 23 Total Hemispherical Emittance of Platinum Versus Temperature

Aeronautical Systems Division, Dir/Materials and Processes, Physics Lab, Wright-Patterson AFB, Ohio.
Rpt No. WADD-TR-61-94, Part II. TOTAL NORMAL AND TOTAL HEMISPHERICAL EMITTANCE OF POLISHED METALS. Jan 63, 31p. incl illus.
Unclassified Report

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1. Molybdenum
2. Platinum
3. Normal emittance
4. Spectral emittance
5. Hemispherical emittance
6. Resistivity
I. AFSC Project 7360 Task 736001
II. MIPR 33(616)-61-7
III. U. S. Naval Radiological Defense Lab., San Francisco, Cal.
IV. G. L. Abbott, N. J. Alvares, W. J. Parker
V. Aval fr OTS
VI. In ASTIA collection

Aeronautical Systems Division, Dir/Materials and Processes, Physics Lab, Wright-Patterson AFB, Ohio.
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